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Report 2463

Differential Scanning Calorimetry as a Method for Indicating Hydrolysis of Urethane Elastomers

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Report Date: June 1988

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Polyether and polyester urethane elastomers were aged in humidity (98%), water, and air at 160°F, 170°F, and 185°F for 7, 14, 28, 42, 70, and 100 days. After each aging condition, tensile strength of the materials was determined and Differential Scanning Calorimetry (DSC) traces were run to observe changes in thermal transitions of the aged specimens. An effort was made to relate these transitions with actual reduction in tensile strength. Results were evaluated using DSC as a method for indicating hydrolytic degradation of the urethane elastomers studied in this investigation.

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PREFACE

This report details a study of the hydrolysis of polyether and polyester urethane thermoplastic elastomers. The Differential Scanning Calorimeter (DSC) was used to examine the polyurethanes after each aging condition. Changes in the DSC thermograms were observed with respect to the deterioration of the materials' physical properties after aging. An effort was made to correlate the DSC data with that of tensile retention so that the DSC could be used as a method of indicating hydrolytic degradation of the polyurethane elastomers.

Historically, certain polyurethane elastomers are known to undergo severe hydrolytic attack when used in hot humid environments. Simply stated, degradation occurs through the hydrolytic splitting of linkages between the structural units of the chain. When severe hydrolytic attack has occurred, as indicated in the aging studies, the polyester urethane elastomer becomes so embrittled that it crumbles with general handling. The purpose of this study is to determine a method of using DSC that can identify the degree of hydrolytic degradation a urethane elastomer has undergone in terms of its physical integrity. Such information would be used in later studies to develop lifetime expectancies of urethane elastomers exposed to hot wet environments.

CONTENTS

SECTION I. EXPERIMENTAL

Two polyurethane elastomers were evaluated using Differential Scanning Calorimetry (DSC). Although the exact chemistries of the materials were not known, both materials were thermoplastic urethane elastomers reacted with methylene diphenyl diisocyanate (MDI). The materials differed in their soft segment structure, one being a *polyether* and the other a *polyester*. Neither material contained any additional compounding ingredients.

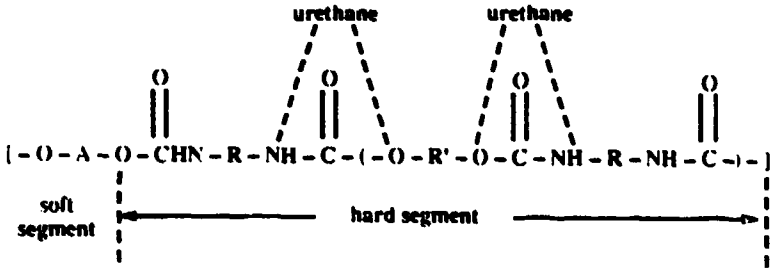
The materials were subjected to: a series of aging conditions in a humidity chamber (98% humidity), immersion in distilled water, and dry oven aging. Samples from each environment were evaluated after 7, 14, 28, 42, 70, and 100 days at 71 °C (160 °F), 77 °C (170 °F), and 85 °C (185 °F). DSC traces were run on each sample unaged and, after each aging condition, tensile retention was also determined.

DSC traces were obtained on a Dupont 9900 Thermal Analysis System using a Dual Sample Cell. Each run was done under a nitrogen purge of 50cc/min. The method involved a ramp of 20 °C per minute to 350 °C. Separate runs were made to determine the glass transition temperature (Tg) of the aged material. These traces were run at 20 °C per minute from -100 °C to 150 °C. Original samples were run from -100 °C to 450 °C to determine both the Tg and degradation temperature.

The tensile data on the humidity samples was obtained from samples prepared from test sheets according to the procedure described in ASTM D412, *Standard Test Methods for Rubber Properties in Tension*. The samples were aged in a temperature and humidity chamber at 98% humidity for the previously described time intervals. After each aging period, the breaking strength was determined using the Scott Tensile Testing Machine Model CRE/1000 at a crosshead speed of 20 inches per minute. The air and water aged specimens were tested on the Instron Testing Machine Model 1125 using the same method as described above.

SECTION II. CHEMISTRY

Before proceeding with the results of this investigation (Section III), this section presents basic urethane chemical structure. As stated earlier, the exact chemistries of the materials studied were not known because they were supplied by the manufacturer on a proprietary basis. However, it was known that both materials were thermoplastic urethanes— one reacted with polyether and MDI, and the other reacted with polyester and MDI. Neither material was compounded with any anti-degradation agents. The basic chemistry of the two urethanes investigated is shown below.[1]



The diagram shows that the soft/flexible segment is attributed to the polyester or polyether portion, whereas the urethane/isocyanate portion compose the rigid/hard segments of the polymer chain. The rigid segments are held together by hydrogen bond interactions in partially ordered domains generally described as *paracrystalline*.

SECTION III. RESULTS AND DISCUSSION

DSC traces of the urethanes characterized each polymer as follows. The polyether urethane exhibited a Tg associated with the soft segment at approximately -43 °C. This transition was followed by two distinct endotherms at 83 °C and 154 °C. It is believed that the first of these endotherms was associated with dissociation of the hard and soft segments or softening of the hard domains, while the second peak was associated with the melting of the paracrystalline domains or bond dissociation within the hard segments. Complete dissociation of the material occurred around 373 °C.

The polyester urethane exhibited a Tg of -20 °C, followed by two endotherms at 61 °C and 120 °C. Complete dissociation of this material occurred at 354 °C. DSC traces showing these transitions of unaged polyether and polyester urethane are shown in Figures 1 and 2.

The most notable change in the DSC traces after aging was an upward shift in endothermic peak temperature associated with the softening and melting of the hard domains. While this trend was seen after aging in water, humidity, and air, these shifts were more pronounced and occurred earlier in the aging cycle when the material was exposed to moisture.

These upward shifts in peak temperature were most notable after aging 28 days in humidity and water at 71 °C (160 °F), and after 7 days at 85 °C (185 °F) predominantly in the ester material. After 28 days in humidity at 71 °C (160 °F), the ester material retained 39% of its tensile strength, while the ether material retained 76%. Tensile retention of the ester urethane dropped to 15% after 14 days in humidity at 85 °C (185 °F), while the ether retained 57% tensile at the same condition. Tensile retention data for both materials aged in humidity and water are shown in Figures 3 and 4.

Figure 5 shows the DSC traces of both materials after humidity aging for 28 days at 77 °C (170 °F). The ether urethane retained 60% tensile strength, while the ester material completely disintegrated, exhibiting no retention of tensile.

The DSC traces show that the ether material exhibited a shift in endothermic peak temperature to 129 °C and 160 °C, and the ester material exhibited an increase in peak temperature to 135 °C and 148 °C. The shift in endothermic peak temperature of the ether urethane was still below that of the paracrystalline melt of the unaged sample which was 160 °C. However, both endothermic peaks of the ester material exceeded the unaged paracrystalline melt of 120 °C. This upward shift in endothermic peak temperatures resulted due to re-ordering and phase separation between the hard and soft segments. This was also seen in the samples that were aged in dry air ovens, discussed later in this report.

After aging in humidity 7 days at 77 °C (170 °F), both materials showed good tensile retention. The ester material retained 100% tensile strength and the ether material exhibited 80% tensile retention. DSC traces of both materials after this aging condition are shown in Figure 6. These traces show slight but not significantly reduced Tg's for each material, and a very distinguished endotherm at 100 °C most likely due to the vaporation of absorbed water in the material. This transition indicated that absorbed water was reversible at this aging stage, leaving the material only slightly affected by hydrolysis.

This observation is explained in Mendelsohn and Rosenblatt's paper.[2] Their research showed that early exposure of polyurethane to heated water resulted in a reversible absorption of water and a slight advancement of cure. It is believed that in these early stages the accelerating affect of the heat and the unreacted isocyanate groups and water provided further chain extension. At this early exposure to heated water, such reactions are believed to be present to a greater degree than hydrolysis.

However, as the hydrolysis proceeded further, it is believed that the urethane linkages are cleaved, resulting in a reduction in the size of the polymer chain. Continuing hydrolysis resulted in increased concentrations of absorbed water, a reduction in Tg, and further increased in endothermic peak temperatures. This was observed in the ester urethane after 28 days of aging in humidity at 77 °C (170 °F). DSC results showed that the aging conditions caused phase separation of the hard and soft domains in both urethane materials. This is described by the increase in endothermic peak temperatures. It is reasonable to assume this separation of phases exposes the soft segment of the polymer to the effects of water. Hydrolysis of a polyester urethane, as discussed in Hepburn,[1] cleaves the polymer chain in the ester portion breaking down the material to acid and glycol products which further accelerate the degradation process. The phase-separated polyester urethane exposes the soft ester segments to the effects of water thereby making the material more susceptible to hydrolytic degradation. Although the ether urethane also undergoes this phase separation, hydrolysis of the ether urethane cleaves the chain at the urethane linkage leaving the soft segment of the material in tact.[1]

Figure 7 shows the DSC traces associated with the materials aged 28 days in humidity at 77 °C (170 °F). The tensile retention of the ether and ester urethanes after this aging condition was 60% and 0%, respectively. Although the DSC traces of both materials looked similar, the ester material showed a drastic change in Tg from -20 °C to -38 °C, as well as an endotherm just above 0 °C due to the melting of water in the sample indicating an irreversible saturation of absorbed water. The ether materials also exhibited this endotherm; however, the Tg of the ether material had changed only slightly from -43 °C to -47 °C, and the endotherm was half the magnitude of the ester urethane indicating a lesser degree of saturation.

Figure 8 shows a DSC trace of the ether material after 100 days of aging in humidity at 77 °C (170 °F). Tensile retention of the ether material at this stage of aging was only 47%, yet the Tg of the material was essentially unchanged as well as the presence and magnitude of the endotherm close to 0 °C. Figure 8 also shows the ester material after 42 days of water immersion at 71 °C (160 °F). The ester material was almost completely

degraded at this aging condition exhibiting only 12% tensile retention. There was no Tg detectable in this DSC trace. There was a large endotherm at 11 °C that again indicated complete saturation of the material with water. The drastically changed Tg of this material, having undergone significant hydrolysis, leads one to believe that the mechanism of hydrolysis in polyester urethanes reduces the soft segment of the polymer to the degree that it is no longer detectable in its original form. It is believed that the endotherm associated with the melting of absorbed water occurs slightly above 0 °C because the water is probably bound to the molecule and may have restricted mobility. Although we feel that this endotherm is associated with the melting of absorbed water, it has been suggested that it may also be related to the melting of the soft segment of the polymer.

However, when the soft domain of the polymer was still present, even though its tensile retention was only 47%, the DSC trace did not indicate significant changes. The tensile retention of the ether material after 28 days in humidity at 77 °C (170 °F) was 60%, while after 100 days, the material retained 47% tensile strength. The DSC traces after these aging conditions were essentially the same as shown in Figures 7 and 8. Here, the only change in DSC data was a shift in the first endothermic peak from 127 °C at 28 days to 138 °C after 100 days. Although both peak temperatures were below the paracrystalline melt of 160 °C, an upward trend in the endothermic peak temperature for the ether material after humidity aging did correlate with a continuous reduction in tensile properties. This trend was also seen in the ester material but, due to the accelerated hydrolysis of the ester urethane, the shifts in peak temperature were more extreme and did not follow a constant trend as seen with the ether urethane. The changes in endothermic peak temperature after humidity and water aging are shown in Figures 9 and 10.

This data leads one to presume that the Tg, being related to the soft segment of the polymer, will remain relatively unaffected as long as the material retains some degree of elasticity as indicated by the tensile retention of the material. The Tg is grossly affected in the polyester material after 28 days in humidity at 170 °F, and 42 days immersed in water at 71 °C (160 °F), but in both cases the material had no physical integrity and crumbled upon handling. In the case of the ether material, the only indication of DSC data that degradation from 60% to 47% retention had occurred was by further increased endothermic peak temperature, most likely due to reduced presence of the soft segment in the hard segment domains.

As a reference for the hydrolysis study, samples were also aged 7 to 100 days at 71 °C (160 °F), 77 °C (170 °F), and 85 °C (185 °F) in dry air ovens. In terms of tensile retention, the results from this study were quite different from the hydrolysis. After some aging conditions, both urethanes showed increased tensile strength over the original unaged material, as shown in Figure 11.

An upward shift in endothermic peak temperature was also seen in the oven aged samples; however, this shift was very gradual in comparison to the humidity and water immersed samples, as shown in Figure 12. In most conditions the first endothermic peak temperature remained below the paracrystalline melt of the hard domains. The

improved physical properties of the air aged samples was attributed to annealing the materials at a temperature below the melting temperature of the hard domains. The annealing process allowed sufficient mobility in the hard blocks for re-ordering to occur, thereby improving the structure and increasing the degree of crystallinity. Since the highly ordered more crystalline material softens and melts at higher temperatures, an upward shift in endothermic peak temperature was observed.

When the urethane elastomer—especially the polyester urethane—underwent hydrolysis, chain scission occurred resulting in reduced molecular weight and a corresponding reduction in physical properties. As discussed in Hepburn,[1] the hydrolysis of a polyester urethane resulted in a breakdown of the polymer chain into four main portions: a substituted urea, an amine, an acid, and a glycol.[1] The acid and glycol portions indicated the degradation of the polyester segment. It is not known whether Hepburn was describing the hydrolysis of the same urethane structure as evaluated in this study. It is believed that this mechanism of hydrolysis would apply; however, further research is needed to confirm this assumption. Assuming that the material is degrading as described by the above hydrolysis, this does support the extreme change in Tg of the polyester after humidity and water aging, shown in Figures 7 and 8. It appears here that the hydrolysis proceeded to the point that the soft segment was completely reduced. The brittle characteristics of the aged material is explained by the absence of the soft segment.

Hydrolysis of a polyether urethane cleaves only at the urethane bond leaving the polyether portion chemically in tact.[1] Thus, the polyether urethane material exhibited an unchanged Tg throughout the aging studies, even though the material gradually degraded as indicated by the tensile retention. The degradation of this material was most likely due to a reduction in molecular weight due to chain scission at the urethane bond. However, this occurred at a much slower rate than the hydrolysis of the polyether urethane.

Although the physical properties of the air aged, the hydrolyzed samples were very different, and the materials were chemically different after aging, there was a similar trend in the upward shift of endothermic peak temperature for both conditions. For the dry oven aged samples, this transition was known to be caused by the annealing effect of the material as discussed earlier. This trend may also be seen in the hydrolyzed material due to the fact that the soft segment of the material was being reduced or removed from the hard segment domains by the hydrolysis reaction. Leaving the hard segment essentially free of soft segment blocks, the softening endotherm of the hard block would begin to approach its melting temperature, and the separated soft segment would result in a lower Tg or a non-detectable Tg when no soft segment is chemically present, as observed in the aged polyester samples shown in Figures 7 and 8.

SECTION IV. CONCLUSIONS AND RECOMMENDATIONS

In light of this investigation, DSC shows that aging polyether and polyester urethane elastomers in water and air above 70 °C causes an increase in order of the hard and soft domains. While re-ordering generally enhances the structure of the material and increases physical properties in dry environments, it is believed that this phase separation was responsible for the degradation of the material when it was exposed to water. Separation of the hard and soft segments exposed the segments to the effects of water thereby making the material more susceptible to hydrolysis. This was seen most dramatically in the polyester urethane, as the material was cleaved in the ester portion of the chain. Because hydrolysis of a polyester urethane generates acid as a product, the degradation process is catalyzed as hydrolysis proceeds. Thus, a sudden drop in physical properties of the polyester urethane was observed.

The presence of an endotherm at 100 °C was most likely due to vaporization of water indicating that any absorbed water is reversible at this stage. An endotherm at 0 °C appears to be related to absorbed water that is irreversible indicating that some degree of hydrolysis occurred. The presence of this endotherm and an extreme change in Tg relates to a complete loss of physical properties as observed in the polyester urethane after 28 days in 98% humidity at 77 °C.

The polyester urethane maintained some degree of retention of properties throughout the aging study. Also, this material did not exhibit a change in Tg, although it did undergo phase separation as indicated by the upward shift in endotherms. The polyether urethane is not as susceptible to hydrolytic degradation as the polyester urethane. Hydrolysis of the ether urethane cleaves the urethane linkage and not the soft ether segment. The polyether urethane maintained an unchanged Tg throughout the aging study. It is believed the Tg remains unchanged because the soft segment of the polymer chain is not cleaved during the hydrolysis of polyether urethanes. Also, the ether urethane does not undergo a catalyzed hydrolysis; therefore, this material exhibits a much slower rate of degradation. Although polyether urethane is inherently less susceptible to hydrolytic degradation, the phase separation that occurs as a result of aging does play a role in exposing the site of hydrolysis of this material to the effect of water.

The results of this study warrant further research in several areas. Studies need to be done to confirm the actual sites of hydrolysis and degradation products for the materials evaluated in this investigation. Statements made in this paper are based on an assumption that the mechanism of hydrolysis as discussed in Hepburn[1] also applies to materials evaluated in this study.

Further examination of hydrolytically aged urethane elastomers in the soft segment region is recommended. It is apparent through this study that the most relevant changes in the materials' chemical structures will be seen in the soft segment region. The thrust of the investigation detailed in this report focused on analysis above room temperature, thus the upward shift in endothermic peak temperature is observed.

In addition to analysis using DSC, further research may be beneficial in observing changes in modulus of hydrolytically aged samples using the Dynamic Mechanical Analyzer (DMA). This method may be more sensitive to the relationship between the physical integrity of the material in terms of modulus changes in the polymer due to aging.

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Footnotes

1. C. Hepburn, *Polyurethane Elastomers*, Applied Science Publishers (New York: 1982), p. 29 and 321.

2. M. A. Mendelsohn and G. B. Rosenblatt, *Effect of Hydrolytic Degradation on Compression and Deflection Characteristics and Hardness of Structured Castings*, American Chemical Society, (Washington, DC: 1979).

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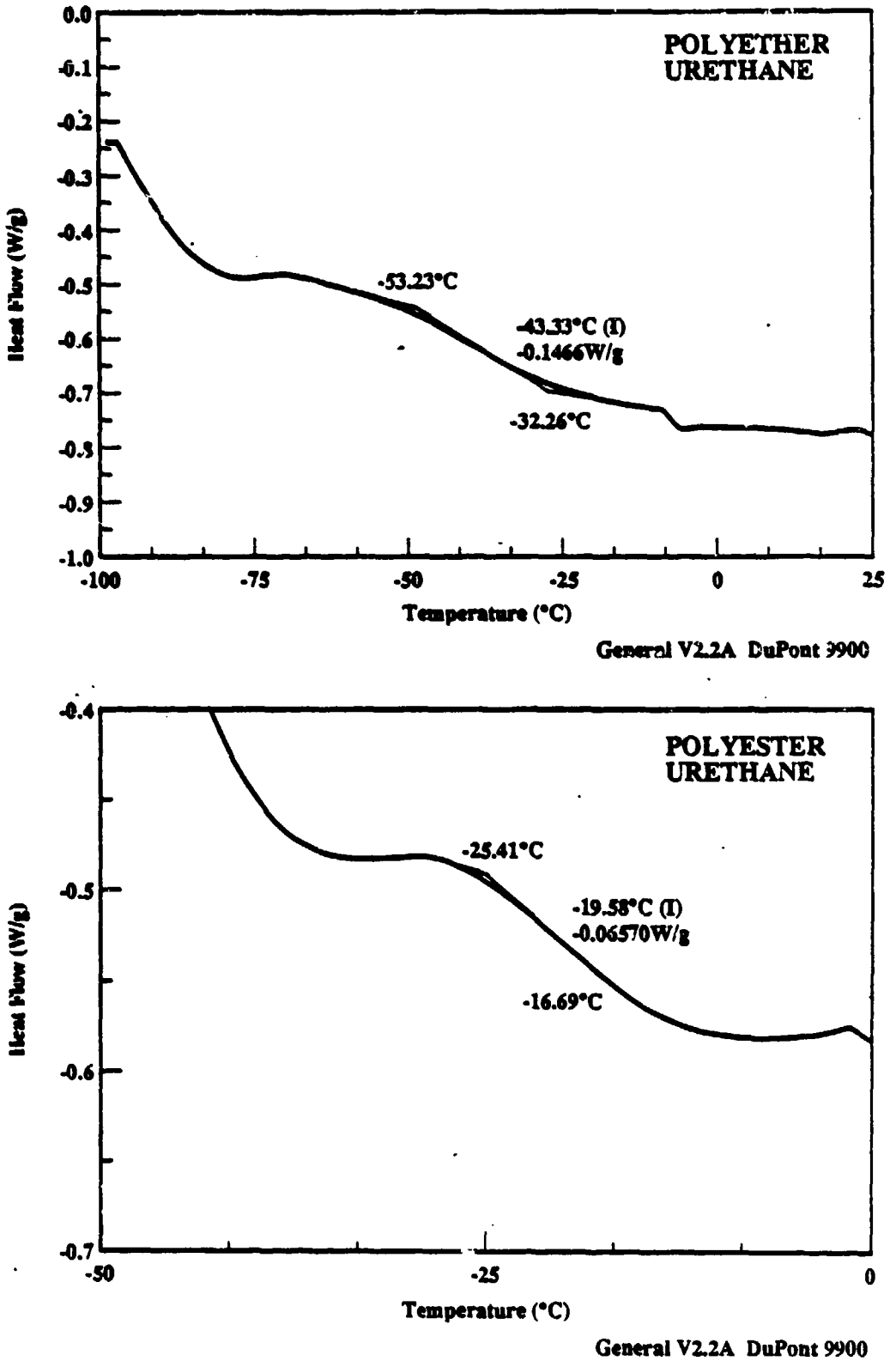
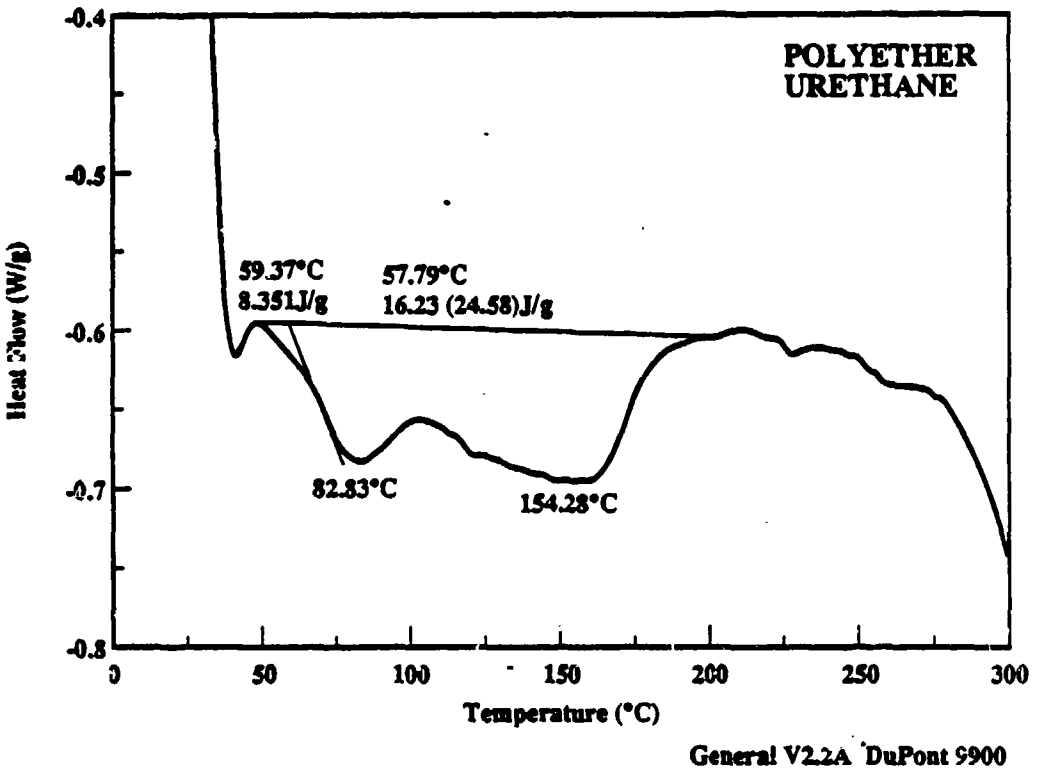


Figure 1. Tg of Unaged Polyether Urethane and Polyester Urethane



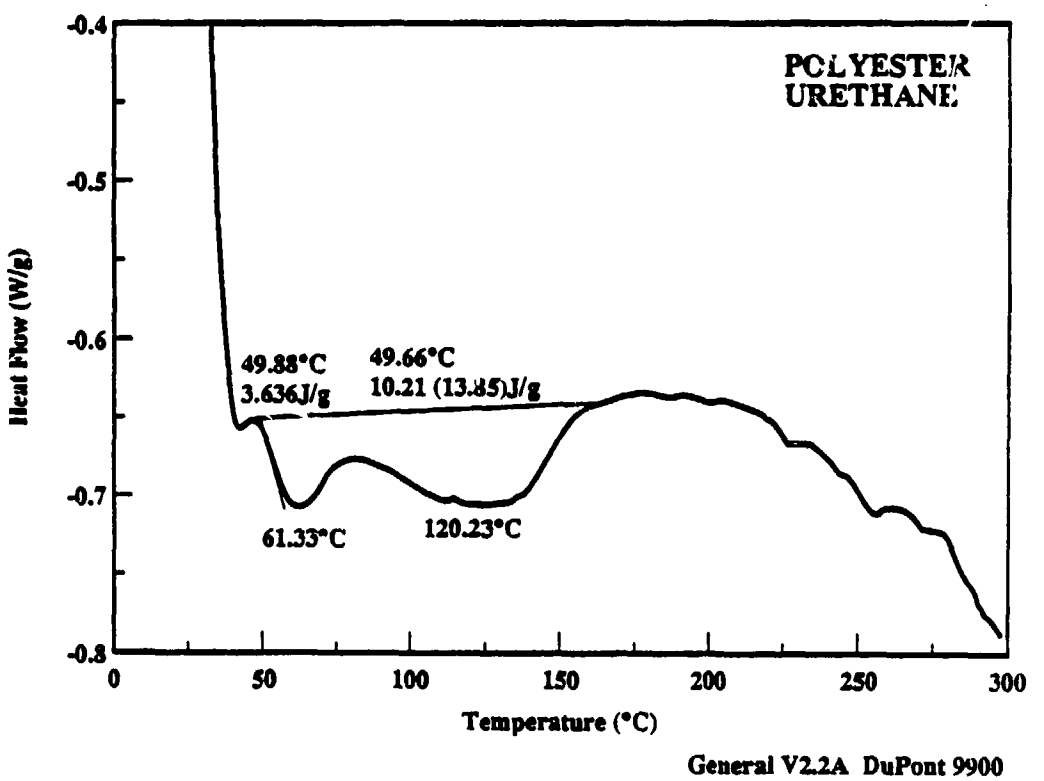


Figure 2. Endotherms of Unaged Polyether Urethane and Polyester Urethane

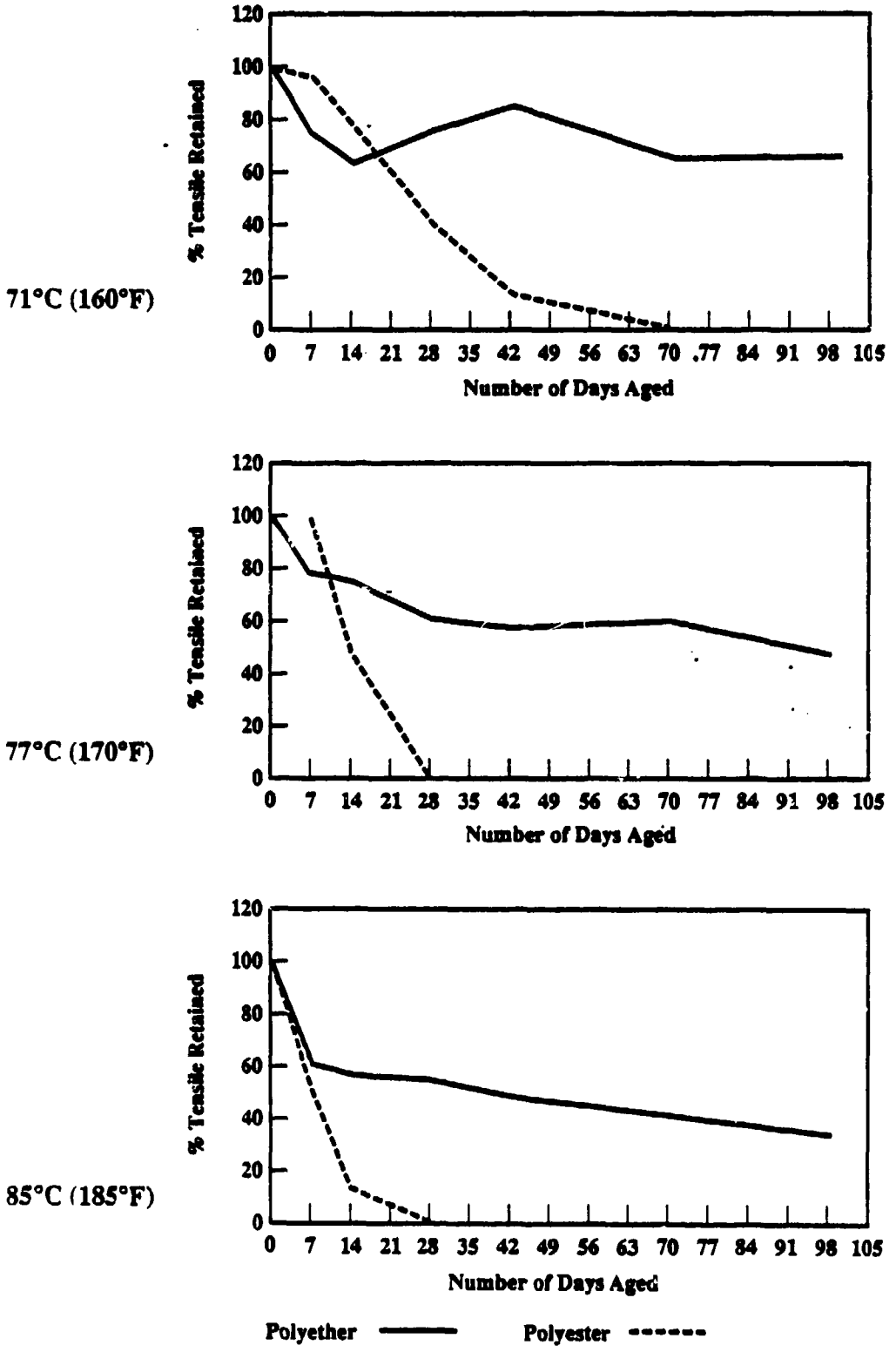


Figure 3. Tensile Retention After Aging in 98% Humidity

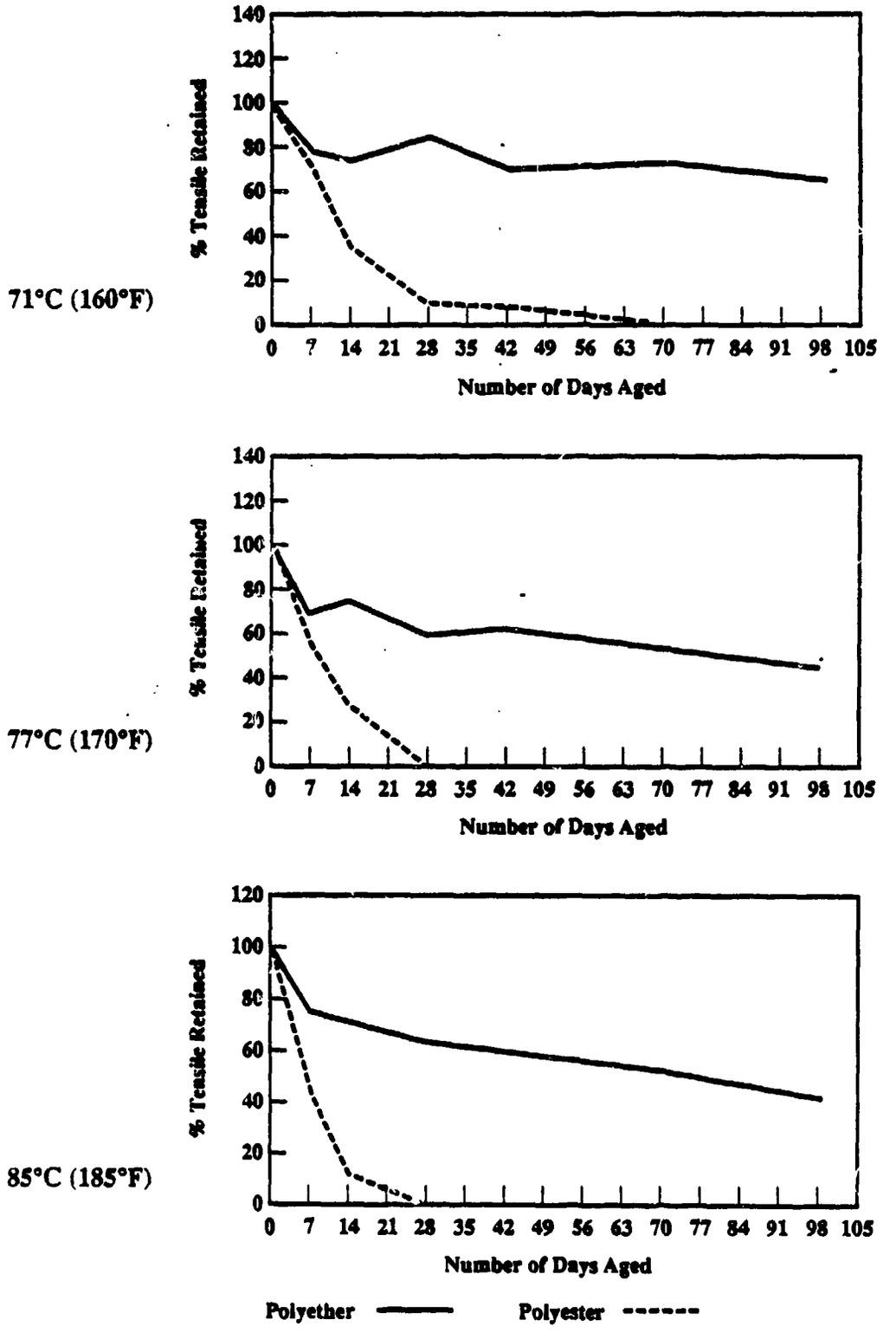


Figure 4. Tensile Retention After Aging in Water

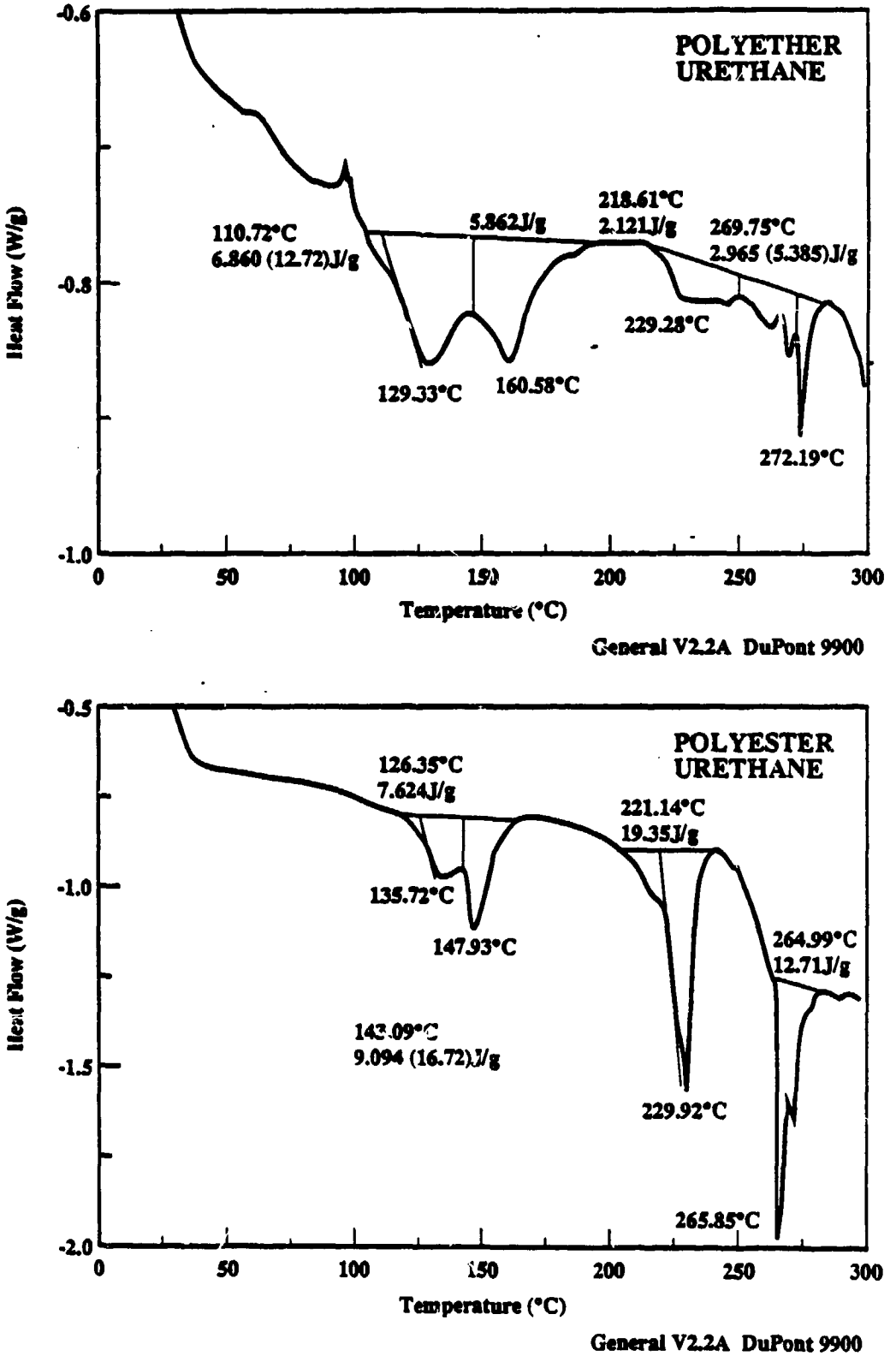
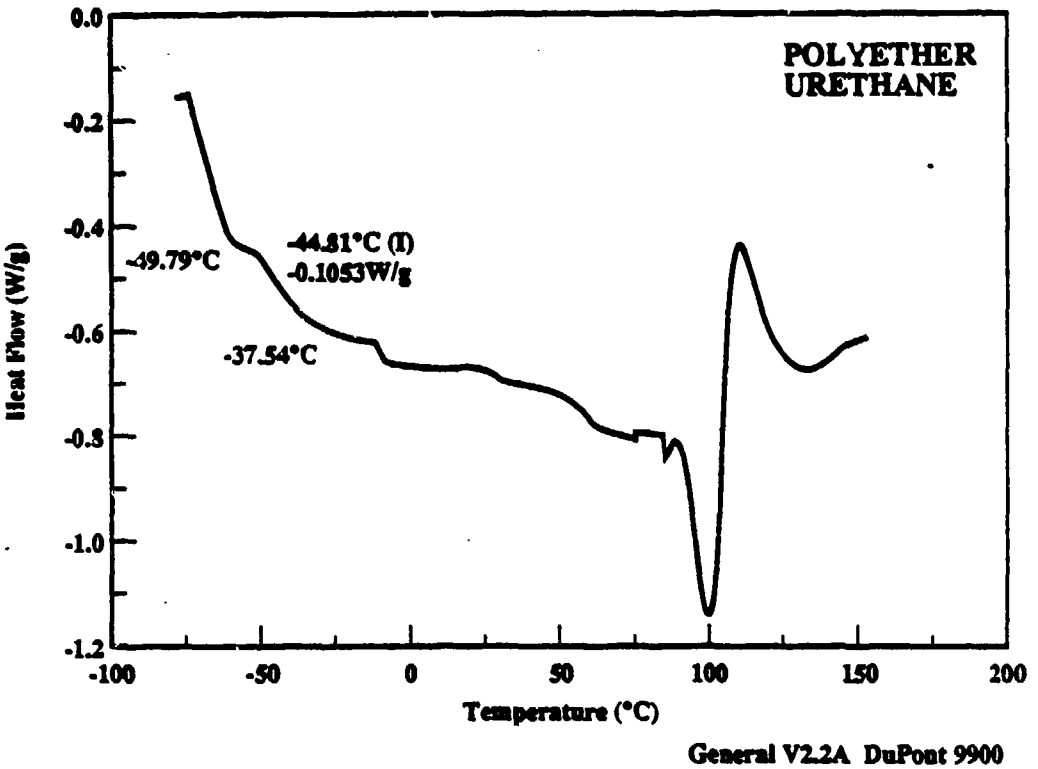


Figure 5. Endotherms After Aging 28 Days in 98% Humidity at 77°C (170°F)



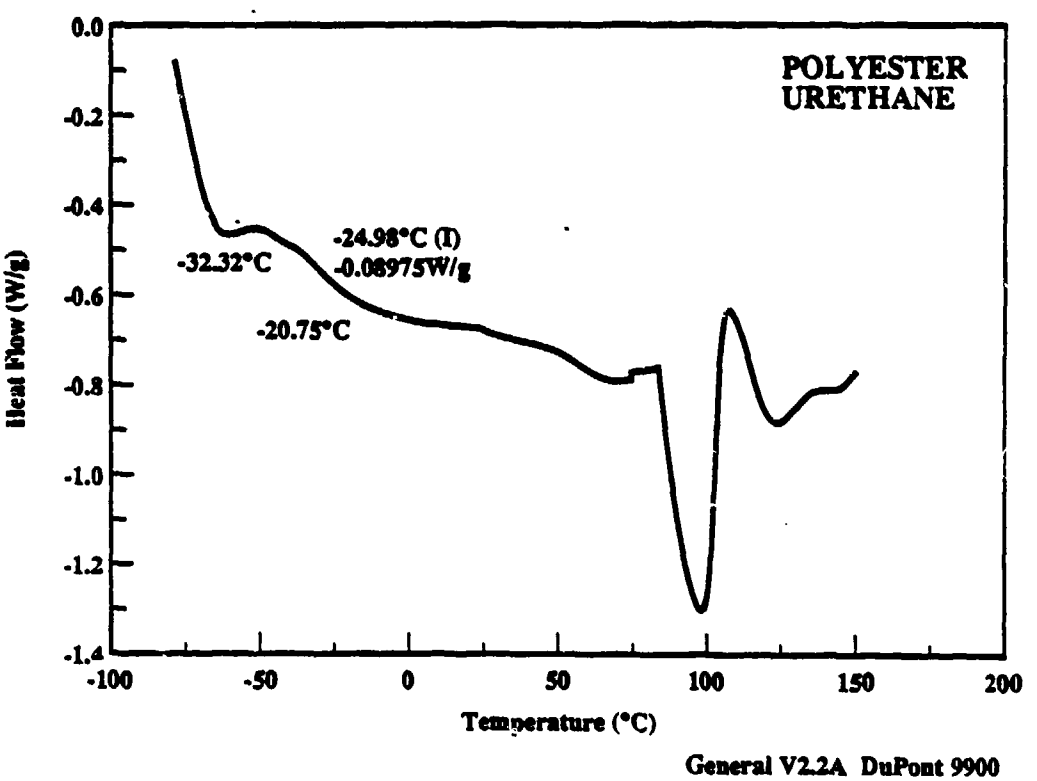
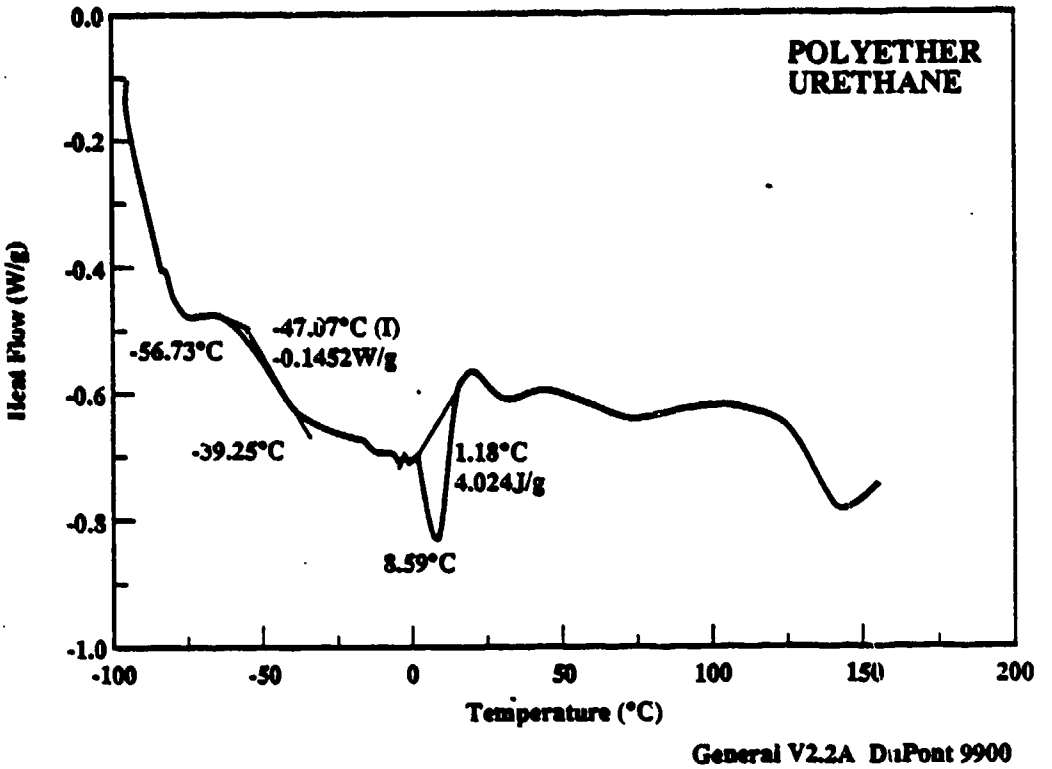


Figure 6. Tg After Aging 7 Days in 98% Humidity at 77°C (170°F)



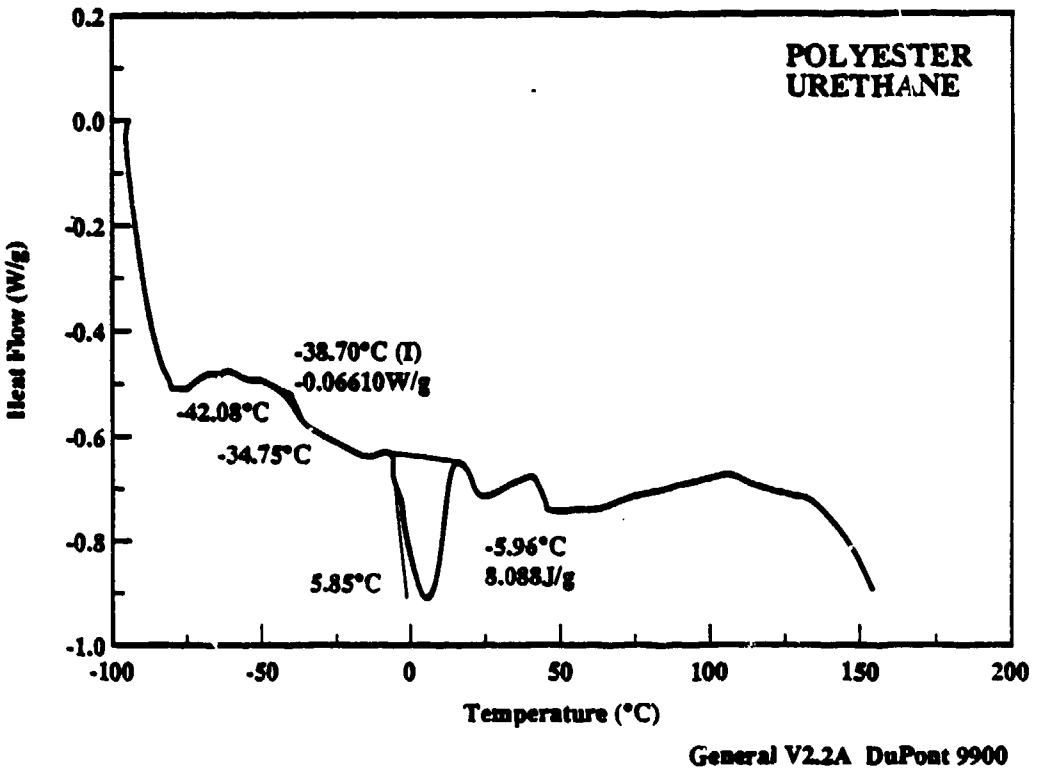


Figure 7. Tg After 28 Days Aging in 98% Humidity at 77°C (170°F)

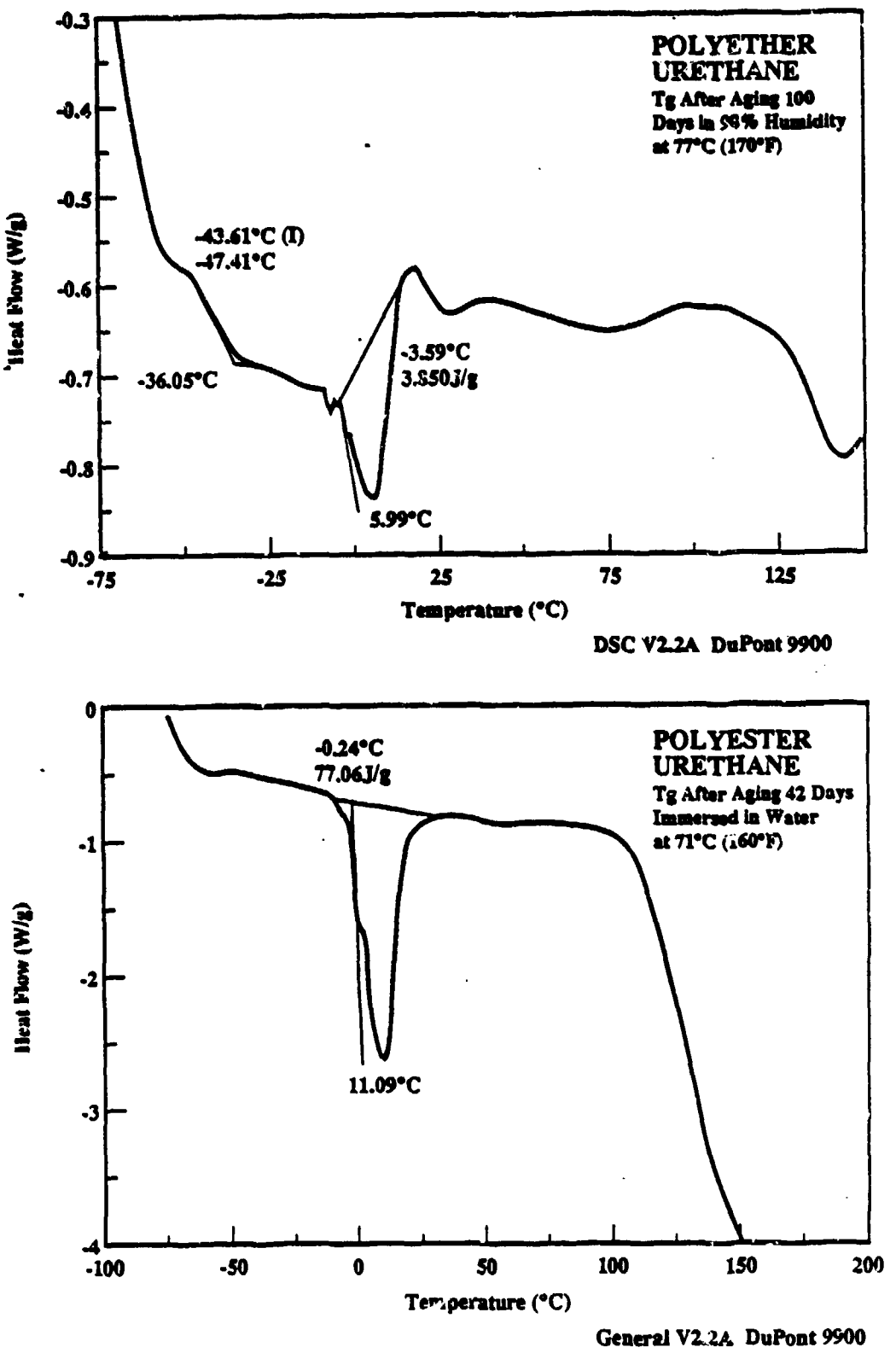


Figure 8. Tg After 100 Days Humidity at 77°C (170°F) and 42 Days Water Immersion at 71°C (160°F)

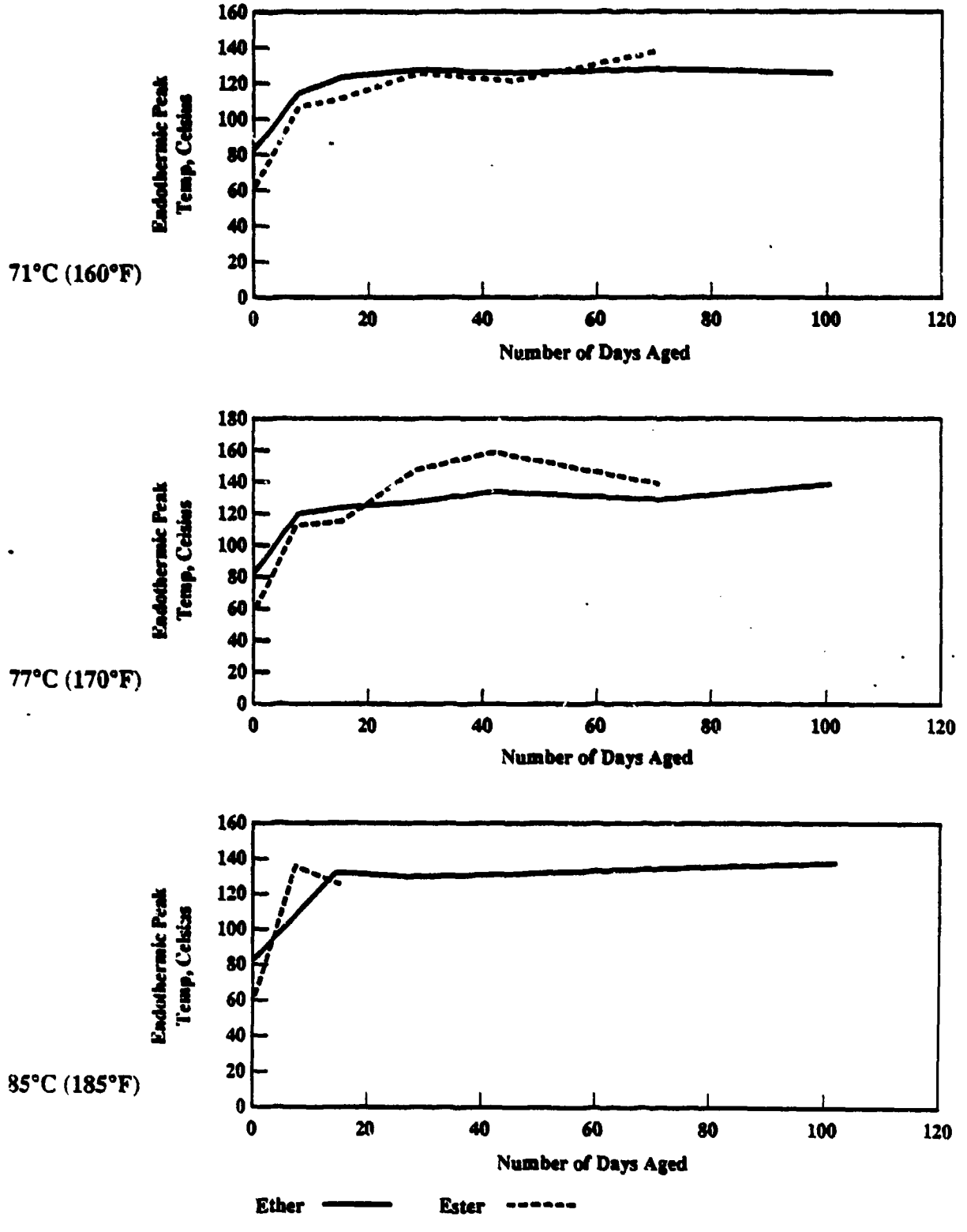


Figure 9. Endothermic Peak Temperatures After Humidity Aging

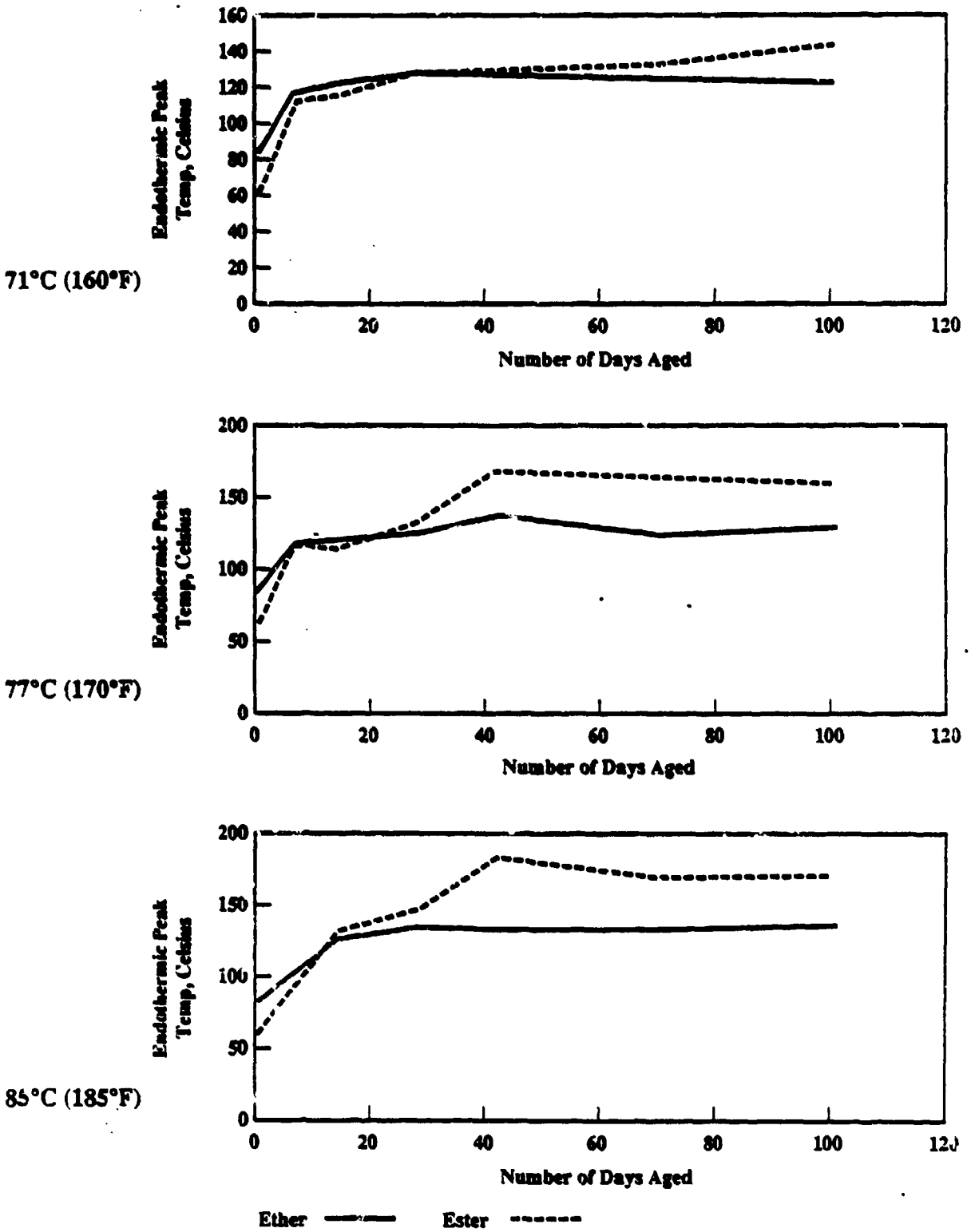


Figure 10. Endothermic Peak Temperatures After Immersion in Water

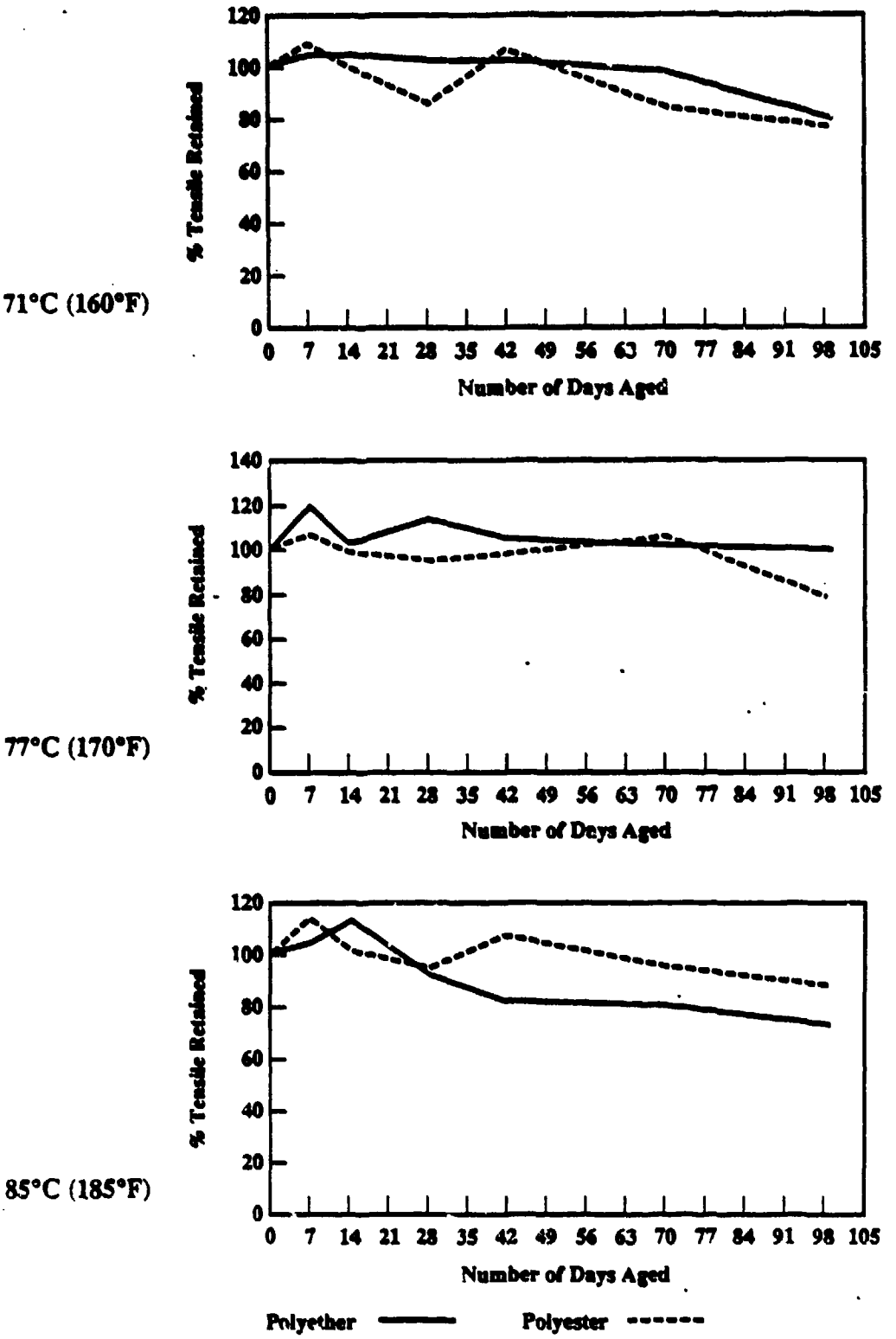


Figure 11. Tensile Retention After Dry Air Aging

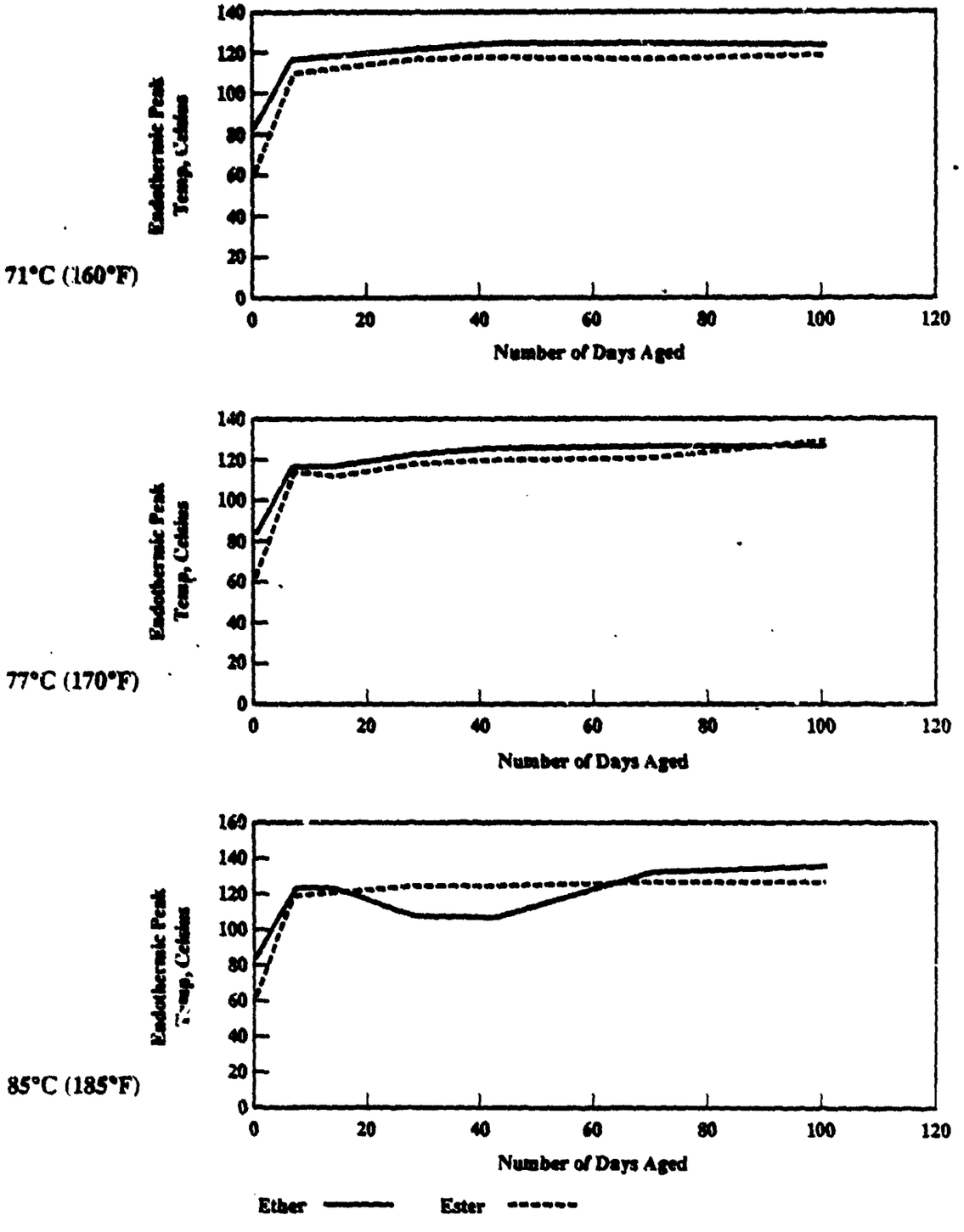


Figure 12. Endothermic Peak Temperature After Dry Air Aging

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